Washing and Rubbing Fastness of Electroless Plated Polyester Conductive Fabric

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With increasing of electromagnetic pollution and wide use of commercial and military products, there is an increasing interest in electromagnetic interference (EMI) shielding. For EMI shielding materials, typical metals have been used. Electroless copper plating of polyester fabrics was demonstrated in the present investigation and compared to sample coated by Cu-Ni-P plated after five times washing and rubbing fastness test . In order to evaluate the residual copper after applying rubbing the surface of samples, SEM images and WDX analysis have been used. The evaluation of electrical conductivity after washing and rubbing tests shows high stability in spite of detaching of some part of coating which remarkably confirms the high stability in EMI shielding effectiveness.

Keywords: electroless, copper-coating, polyester fabric, washing and rubbing tests, surface resistance

In recent years, there is an increasing interest in electromagnetic interference (EMI) shielding as use of military, scientific electrical products, electronic and commercial devices [1]. In addition, shielding to electromagnetic interferences (EMI) becomes an important concern to our daily health by the symptoms of languidness, headache, insomnia, nervousness, etc. on exposure to electromagnetic fields [2-6].

For EMI shielding materials, typical metals such as copper, aluminum and silver have been used, which have high conductivity [7-9]. To produce materials with high EMI shielding efficiency, various metal coating techniques have been suggested and commercially used as metal foils and laminates, conductive paints and lacquers, sputter coating, electroless plating ,etc. Among them, electroless plating is a promising way to produce metal-coated fabrics[10-11]. This technique has advantages such as homogeneous metal deposition, excellent conductivity , high EMI shielding efficiency , applicability to non-conductors and complex shaped materials[12]. Electroless plating uses a redox reaction to deposit metal on an object without the passage of an electric current, because it allows a constant metal ion concentration to bathe all parts of the object. It deposits metal evenly along edges, inside holes and over irregularly shaped objects, which are difficult to plate with electroplating. Electroless plating is also used to deposit a conductive surface on a nonconductive object to allow it to be electroplated [13-15]. Electroless technologies have been used for many decades. They involve reduction of a complex metal using a mild reducing agent, typically formaldehyde [16].

Because of the high conductivity of copper, electroless copper plating is currently used to manufacture conductive fabrics. Traditionally, electreoless copper plating use formaldehyde as reduce agents. These systems are typically operated at *p*H values above 11 and this bath may release hazardous gases during operation. Therefore, electroless copper plating using sodium hypophosphite as the reducing agent in place of formaldehyde is attractive, because of its low *p*H, low cost, and relative safety features [17-18]. Several papers have studied the electroless copper solutions using sodium hypophosphite as reducing agent.[19]However, the hypophosphite-based electroless copper plating process is complicated ,because copper is not a good catalyst for the oxidation of hypophosphite resulting in little or no plating on a pure copper surface. One approach to catalyze the oxidation of the reducing agent is to add nickel ions (or other metal ions) to the bath, resulting in a very small amount of co-deposited nickel in the copper deposit. The nickel serves to catalyze the oxidation of hypophosphite enabling continuous copper deposition [11,20] .Therefore, electroless copper plating using hypophosphite as the reducing agent is actually Cu–Ni–P alloy [21].

In this paper, we described the electroless plating of Cu–Ni–P alloy on polyester fabrics using sodium hypophosphite as reducing agent and evaluation after five times washing and rubbing fastness test, surface characteristics of copper-plated cotton fibers were investigated in details through the measurements of scanning electron microscopy (SEM) and WDX analysis. Then, the surface resistance(R_S) was investigated too.

Experimental part

Materials and methods

Polyester fabrics (53×48 count/cm^2, 100 g/m^2, taffeta fabric) were used as substrates. The size of each specimen was 10cm × 10 cm.

The chemicals used for the electroless copper plating included nonionic detergent, $NaHCo_3$, $SnCl_2$, HCl, $PdCl_2$, $CuSO_4$, $NiSO_4.6H_2O$, $NaH_2PO_2.H_2O$,H_3BO_3, $K_4Fe(CN)_6$, $C_6H_5Na_3O_7.2H_2O$ were used to electroless plating process. All chemicals were purchased from Merck and used without any further purification.

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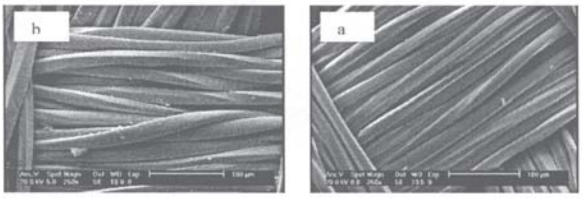

Fig. 1.SEM photographs of the (a)untreated polyester fabric (b) Cu-Ni-P plated polyester fabric

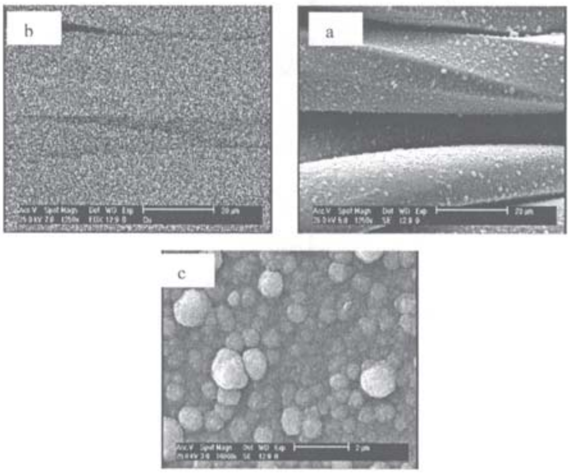

Fig. 2.(a)SEM photograph with 5,000 x magnification (b) WDX analysis (c) SEM photograph with 10,000 x magnification of the Cu-Ni-P plated polyester fabric

Electroless plating was carried out by multi-step processes including scouring, rinsing, sensitization, rinsing, activation, rinsing, electroless polyester plating, rinsing and drying. The fabric specimens (10cm × 10 cm) were first scoured in non-ionic detergent (0.5g/L) and $NaHCo_3$ (0.5g/L) solution prior to use. The specimens were scoured in 10 g/L NaOH solution at70°C for 3 min prior to use. The samples were rinsed with distilled water and etched in a mixture of 15 g/L $KMnO_4$ and 40 mL/L H_2SO_4 solution for 3 min. Then, the samples were rinsed with distilled water. Surface sensitization was conducted by immersion of the samples into an aqueous solution containing $SnCl_2$ and HCl (40mL/L 38% w/w). The specimens were again rinsed with distilled water and immersed in an activator containing $PdCl_2$ and HCl (20 mL/L, 38% w/w). The specimens were then rinsed in a large volume of deionize water for 10 min to prevent contamination the plating bath. Then, all samples immersed in the electroless bath containing copper sulfate, nickel sulfate, sodium hypophosphite, sodium citrate, boric acid and $K_4Fe(CN)_6$. The samples were rinsed with distilled water, ethylalcohol and dried in oven at 70°C.

Scanning electron microscope (SEM, XL30 PHILIPS) was used to characterize the surface morphology of deposits. WDX analysis(3PC, Microspec Ltd., USA) was used to exist metallic particles over surface copper coated polyester fabrics. The EMISE of fabrics were measured by using ASTM D4935-99 technique. Color changing under different application conditions for two standard testing methods, namely, (1) ISO 105-C06:1994 (color fastness to domestic and commercial laundering, (2) ISO 105-A02:1993 (color fastness to rubbing) were used for estimate.

Results and discussions

In order to study the surface characteristics, copper-plated polyester fibers were investigated in details through the measurements of the scanning electron microscopy (SEM) and WDX analysis. Scanning electron microscopy images (SEM) of the untreated and Cu-Ni-P-plated polyester fabric are shown in figure 1 with magnification of 250x. As you can see in the image , the surface of fibers is coated fully and uniformly with a layer of copper metal .So that, the microscopic structure of coating polyester has completely changed.

Figure 2, SEM images of coating samples with 1250 x magnification, the WDX analysis and SEM images of coating samples with 10,000 x magnification are shown. According to figure 2, coated fabric using metal particles leads to formation of a very uniform layer of metal nanoparticles in the range of 20-100 nm are also dense and adhesion of particles to each other, cause electrical conductivity of fabric.

Evaluating of the electrical resistance in the sample surface of copper-plated polyester fibers, indicates electrical conductivity on the sample form and voltage changes applied on Cu-Ni-P plated polyester fabric current average is shown in figure 3. The results indicate aspects of voltage change with an acceptable approximation, almost linear relationship with the intensity of electric current is applied and electrical resistance of the fabric surface is fixed according to the coating of all the fabric surface pores without the need for uniform the electric current coverage , which lead to producing very low of fabric resistance and high conductivity. So, the surface resistance measurement of copper-plated polyester fibers was 0.0038(Ω / sq) and the surface electrical conductivity was 2642 (S / cm).

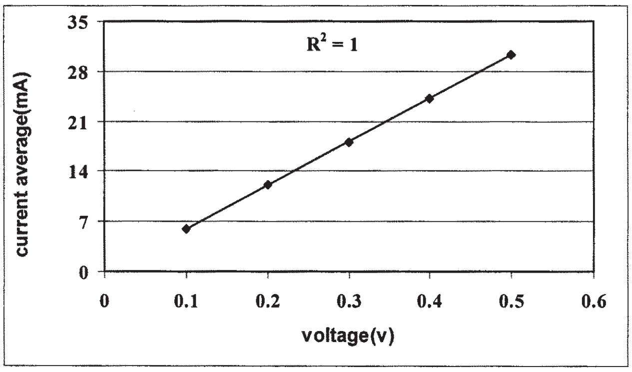

Fig. 3. Changes voltage applied on Cu-Ni-P plated polyester fabric current average

To evaluate the resistance copper-plated polyester fibers, tests related to the washing and rubbing fastness have been evaluated in table 1.

Table 1
EVALUATION OF WASHING AND RUBBING FASTNESS TEST OF THE APPEARANCE

Washing	Rubbing	
	Dry	Wet
5	4-5	3-4

Considerations show that washing does not affect appearance of the color sample, but practice makes wear and color changes of pallor sample more than abrasion wear which is dry. Fabric surface electrical resistance before and after rubbing has been evaluated (fig. 4) .

The results of surface resistance increase 51% in dry rubbing and increase 32% in wet rubbing. Therefore, considerable amounts of copper particles

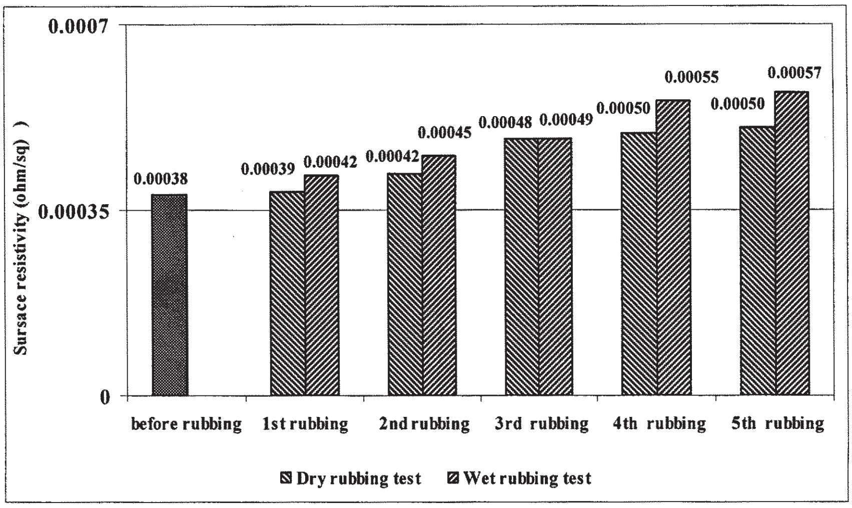

Fig. 4.dry and wet rubbing effect on the conductivity of polyester fabrics with copper coating

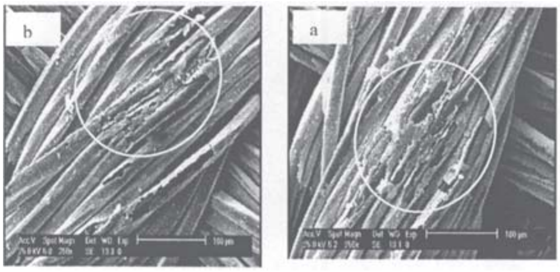

Fig. 4.SEM image taken after rubbing five times of copper-plated polyester fibers a) dry b) wet

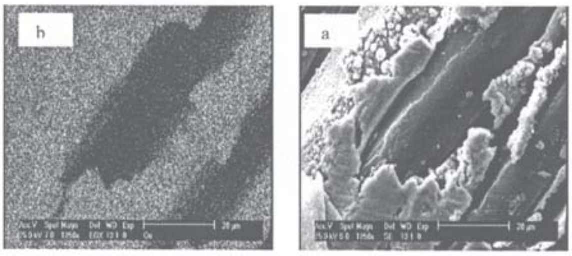

Fig. 5.(a) SEM image after dry rubbing five times b) WDX analysis after dry rubbing five times

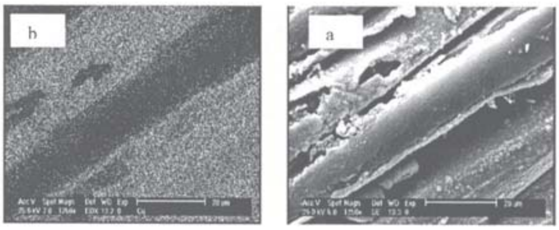

Fig. 6.(a) SEM image after wet rubbing five times b) WDX analysis after wet rubbing five times.

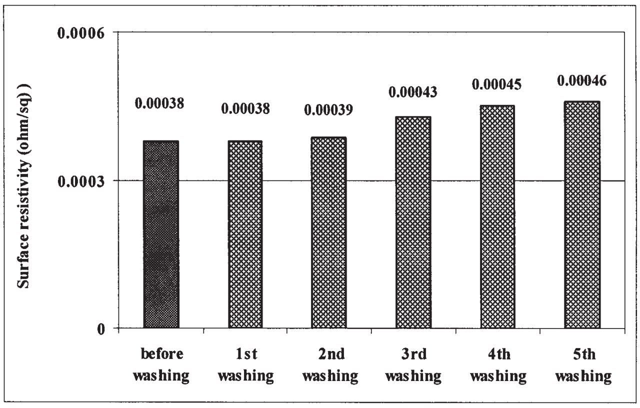

Fig. 7. Washing effect on copper-plated polyester fibers conductivity

are separated from the fabric surface that reduces the thickness and conductivity are formed. In this case, the wet rubbing is more remarkable than dry one. However, the compact and homogenous distribution of conductive particles provide a great conductive coating on fabric surface with high durability, even dry and wet rubbing tests, which caused electrical conductivity in samples. The SEM images of five times dry and wet rubbing tests with 1250 x magnification are shown in figure 4(a,b).

The evaluation of electrical conductivity after rubbing tests shows high stability in spite of detaching of some part of coating which remarkably confirms the high stability in EMI shielding effectiveness. In order to evaluate the residual copper after applying rubbing, surface samples using a WDX system was evaluated. Figure 5(a,b) and figure 6 (a,b), respectively shows the SEM and WDX analysis copper plated surfaces of five times dry and wet rubbing polyester fiber.

Figure 2 WDX analysis indicated reduction copper particles on the surface after applied rubbing. Thus, the fabric surface rubbing is still having electrical conductivity. These results consistent with the results obtained the evaluation of surface electrical resistance before and after the rubbing tests.

In order to evaluate the fabrics conductivity after washing test, the surface resistance of samples after testing has been shown in figure 7.

Figure 5, describes obvious washing effect on low conductivity samples and surface resistance increase 21% after washing. Thus, fewer particles are separated from the fabric with repeating washing. However, this level of reduction in samples conductivity after washing can be ignored.

Conclusions

In this paper, we described the electroless plating of Cu–Ni–P alloy on polyester fabrics. The results show that with applying washing and rubbing test, considerable amounts of copper particles are separated from the fabric surface that reduces the thickness and conductivity are formed. However, the compact and homogenous distribution of conductive particles provide a great conductive coating on fabric surface with high durability, which caused electrical conductivity in samples. In addition, The SEM images and WDX analysis indicated that the deposits became more compact, uniform and smoother, also exist homogenous metal particle distribution over coated fabric surface.

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